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Swelling and Alloy Stability of Potential Fast-Breeder-Reactor Structural Alloys Irradiated in NRL Experiment H-5

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Seven commercial alloys of potential interest for fast-breeder-reactor structural alloys have been irradiated in the EBR-II to a fluence of 1.6×10^{22} n/cm^2, E > 0.1 MeV, at 455°C (850°F) and subsequently examined and analyzed using transmission electron microscopy. Irradiated were Inconel 718, Incoloy 800, Inconel 600, Inconel 625, PE-16, Sandvik 12R72HV, and Inconel 744. The experiment was designed to examine the alloy stability during irradiation and to measure swelling resistance. over
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20. Abstract (Continued)

Inconel 718 and PE-16 exhibited the best swelling resistance, and Sandvik 12R72HV and Incoloy 800 had the poorest resistance to swelling. Inconel 744, an experimental superplastic alloy with a duplex microstructure, exhibited one of the highest swelling rates known for the austenite phase but showed no swelling for the ferrite phase. Inconel 625, Inconel 600, and Incoloy 800 showed extensive precipitation of phases not present prior to irradiation.

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SWELLING AND ALLOY STABILITY OF POTENTIAL FAST-BREEDER-REACTOR STRUCTURAL ALLOYS IRRADIATED IN NRL EXPERIMENT H-5

INTRODUCTION

Swelling and alloy stability may in many cases limit the life of cladding and duct materials. Although the structural components of the fast breeder reactor (FBR) will be subjected to lower fluences than the cladding or duct, dimensional changes resulting from swelling and phase changes under irradiation must still be of concern because of the long service lifetime.

The current experiment consisted of the irradiation of transmission electron microscopy specimens of seven commerical alloys of potential interest for FBR structural alloys and their subsequent examination and analysis. The specimens were irradiated in NRL experiment H-5 in the experimental breeder reactor in Idaho Falls, Idaho (EBR-II reactor), to a fluence of 1.6×10^{22} n/cm^2, $E > 0.1$ MeV, at 455°C (850°F). Materials irradiated were Inconel 718, Incoloy 800, Inconel 600, Inconel 625, PE-16, Sandvik 12R72HV, and Inconel 744. Following irradiation the specimens were examined by transmission electron microscopy (TEM) to determine the swelling resistance using standard practices for TEM measurements. The stability of each alloy under irradiation also was qualitatively assessed, by comparing the microstructures before and after irradiation. A brief commentary on the phases to be expected in each system has been included in the discussion section, so that expected phases can be compared with those observed. No attempt has been made to identify all the phases observed, as this was beyond the scope of the study. Such detailed characterization should be conducted, however, if an alloy is selected for use in FBR.

MATERIALS HISTORY AND EXPERIMENTAL TECHNIQUES

The compositions of the alloys studied in this experiment are given in Table 1. All alloys were obtained from the International Nickel Company except for the Sandvik 12R72HV alloy, which was obtained from Sandvik Steel Company.

Sheet stock of all the alloys was produced by rolling from a variety of dimensions to 1.58 mm (0.062 in.) with intermediate anneals in vacuum. The final reduction was from 1.58 mm to 0.08 mm (.003 in.). Following the final reduction the specimens were heat treated according to the schedules shown in Table 2. After the final heat treatment TEM specimens 3.0 mm (0.118 in.) in diameter were punched from the sheet. These specimens were then encapsulated in a subcapsule of 316 stainless steel backfilled with helium. The specimens were tightly packed with a spacer block to assure good thermal contact with the capsule.

The subcapsule was part of a larger assembly designated NRL experiment H-5 which was irradiated in subassembly X188 in core position 4F2 of the EBR-II reactor. This

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Table 1 — Alloy Compositions

Materials	Composition (wt - %)													
	Ni	Cr	Fe	C	Mn	P	S	Si	Ti	Al	Cb	Mo	Cu	Co
IN 600	76.0	15.0	8.3	0.04	0.19	-	-	0.27	-	-	-	-	0.30	-
IN 625	62.1	21.9	2.7	0.03	0.07	-	-	0.20	0.26	0.14	3.55	9.02	-	-
IN 718	53.1	18.3	18.5	0.01	0.20	0.01	0.005	0.10	0.89	0.59	5.20	3.06	-	-
IN 744	7.8	27.4	Bal	0.055	0.62	0.016	0.007	0.50	-	-	-	-	-	-
IN 800	30.5	20.8	47.0	0.09	0.71	-	-	0.61	-	-	-	0.27	-	-
PE-16	43.2	16.4	34.2	0.05	0.06	-	-	0.25	1.18	1.24	-	3.16	-	0.21
12R72HV*	15.0	15.0	Bal	0.10	1.80	-	-	0.50	0.45	-	-	1.20	-	-

*Nominal composition.

Table 2 — Preirradiation Heat Treatment

Material	Heat Treatment
Inconel 718 Inconel 600 Incoloy 800 PE-16	1200°C for 1 hr; air cooled 790°C for 4 hr
Inconel 625	1200°C for 1 hr; cool rate = 490°C/hr 790°C for 10 hr; cool rate = 650°C/hr 650°C for 10 hr; cool rate = 460°C/hr
Inconel 744	870°C for 1 hr; air cooled 455°C for 3 min; air cooled
Sandvik 12R72HV	1200°C for 1 hr; air cooled 20% cold worked, followed by 850° for 4 hr; air cooled

subassembly received a total fluence of 2.2×10^{22} n/cm^2 at the core midplane. The position of the specimens was 13 cm above the core midplane, where the neutron flux is 0.83 of the value at the core midplane. The ratio of neutron flux with $E > 0.11$ MeV to the total flux at this location is 0.87, so that the neutron fluence for $E > 0.1$ MeV for this experiment was calculated to be 1.6×10^{22} n/cm^2. Flux monitors included in the experiment have not been analyzed yet. The capsule was exposed to flowing sodium coolant, which at this reactor location and power level had a temperature of 455°C ± 25°C (850°F). Following irradiation the capsules were decontaminated and returned to NRL for examination of the specimens.

The specimens were thinned to transparency by electropolishing and examined with a JEM 200A electron microscope equipped with a side-entry goniometer stage. The microscope was operated at 200 kV for all observations. Foil thicknesses were determined from stereo measurements with stereo-pair micrographs taken after tilting the sample symetrically about the X axis 4 to 5 degrees on each side of 0. Foils were typically 0.1 μm thick, and the accuracy of thickness measurements is estimated to be ±20%. Void sizes were measured on photographs enlarged 3X with the aid of a Zeiss particle-size analyzer.

RESULTS

The microstructural changes observed after irradiation in the H-5 experiment are briefly described for each material in the following subsections. These microstructural changes include void formation and changes in the precipitate structure. A summary of the characteristics of the void population and swelling for each alloy is included in Table 3. The phases anticipated from the heat treatment will be described in more detail in the discussion section and will be compared with the observations. In a subsequent section, swelling-composition trends are discussed.

Table 3 — Void Population Characteristics After Irradiation to 1.6×10^{22} n/cm^2, $E > 0.1$ MeV, at 455°C (850°F)

Material	Density (cm^{-3})	Mean Diameter (nm)	Swelling (%)
Inconel 718	5.8×10^{13}	17.3	0.02
Incoloy 800	2.3×10^{14}	26.6	0.26
Inconel 600	1.44×10^{14}	19.6	0.04
Inconel 625	8.0×10^{14}	11.4	0.08
PE-16	6.6×10^{13}	19.4	0.03
Sandvik 12R72HV	2.6×10^{15}	12.7	0.3
Inconel 744 (austenite)	2.8×10^{15}	24.6	2.7
(ferrite)	-*	-	-
(average)	1.0×10^{15}	24.6	1.0

*The ferrite grains did not contain voids.

Inconel 718

Inconel 718 is a nickel-base alloy strengthened by γ'' and γ' precipitates (these phases will be described in detail in the discussion section). These ordered phases are only faintly discernible in bright-field micrographs of the alloy prior to irradiation, and no other precipitates are present.

Examination of the irradiated specimens revealed a low density of homogeneously distributed voids, as can be seen in Fig. 1. The void population had a mean diameter of 17.3 nm and a density of 5.8×10^{13} cm^{-3}, which produced swelling of 0.02%. No obvious changes occured in the precipitate structure, as also can be seen in Fig. 1. The ordered γ'' precipitate structure in the irradiated specimen is shown in Fig. 2 imaged in dark field with a superlattice reflection.

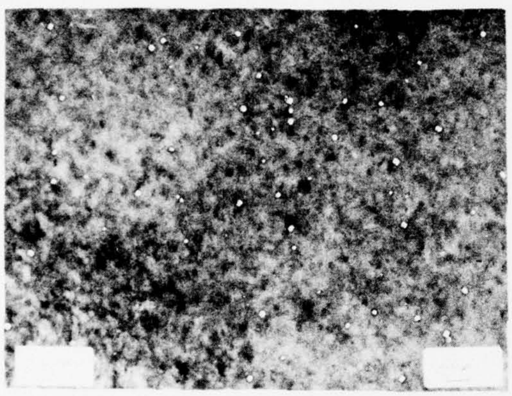

Fig. 1 — Transmission electron micrograph showing void population in Inconel 718 after irradiation to a fluence of 1.6×10^{22} n/cm^2, E > 0.1 MeV, at 455°C (850°F)

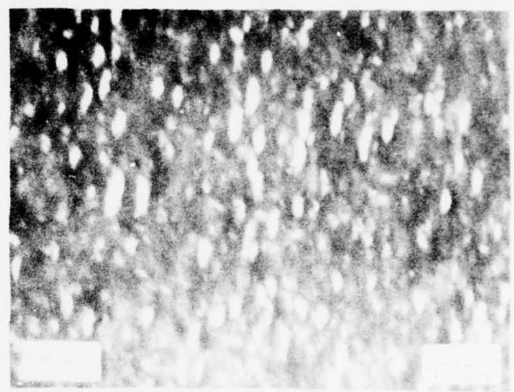

Fig. 2 — Transmission electron micrograph of γ'' precipitates in irradiated Inconel 718 imaged in dark-field contrast using a superlattice reflection

Incoloy 800

Incoloy 800 is a high-nickel austenitic stainless steel which is solution hardened. Prior to irradiation the alloy had only a few precipitates, presumably carbides, along the grain boundary and a few precipitates widely scattered in the grain interior.

Voids in the irradiated sample were distributed homogeneously except for some denuding along the grain boundaries. Figure 3 shows the grain-boundary carbides and the void population both near the grain boundary and in the grain interior. The void population had a mean diameter of 26.6 nm and a density of 2.3×10^{14} cm^{-3}, which produced swelling of 0.26%. Following irradiation, the grain-boundary precipitates appeared to have grown slightly, and a new type of precipitate appeared in the matrix. These precipitates appear to be rod shaped and are preferentially aligned, as can be seen in Fig. 4. The rods are approximately 10 nm in diameter and 40 nm long.

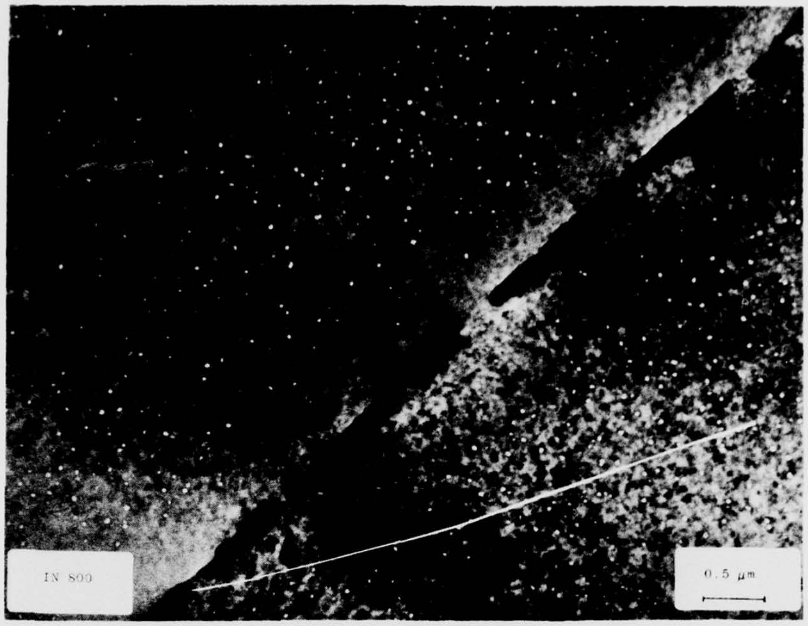

Fig. 3 — Transmission electron micrograph showing grain-boundary precipitates and void population both near the grain boundary and in the grain interior of Incoloy 800 irradiated to a fluence of 1.6×10^{22} n/cm^2, E > 0.1 MeV, at 455°C (850°F)

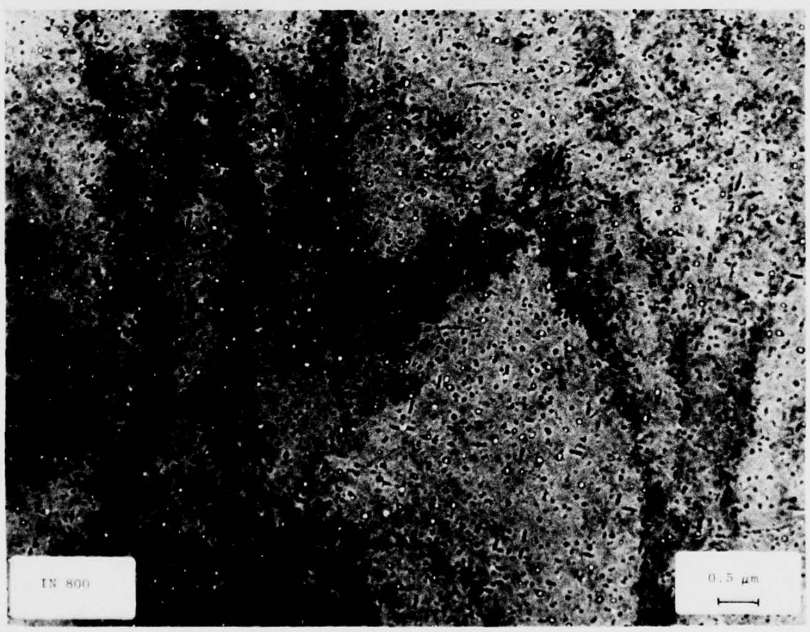

Fig. 4 — Transmission electron micrograph showing high-density of preferentially aligned rod-shaped precipitates in irradiated Incoloy 800

Inconel 600

Inconel 600 is a solution-hardened nickel-base alloy. The unirradiated specimen contained precipitates along a large fraction of the grain boundary, but the grain interior had no visible structure.

Following irradiation, voids with a mean diameter of 19.6 nm and density of 1.44×10^{14} cm^{-3} were distributed homogeneously in the matrix, as can be seen in Fig. 5. Swelling of the alloy was 0.04%. Some denuding of voids along grain boundaries was observed. Numerous dislocation loops with a mean diameter of 37.5 nm formed during irradiation and are illustrated in Fig. 6. A few small precipitates also formed during irradiation and appear as black spots in Fig. 5.

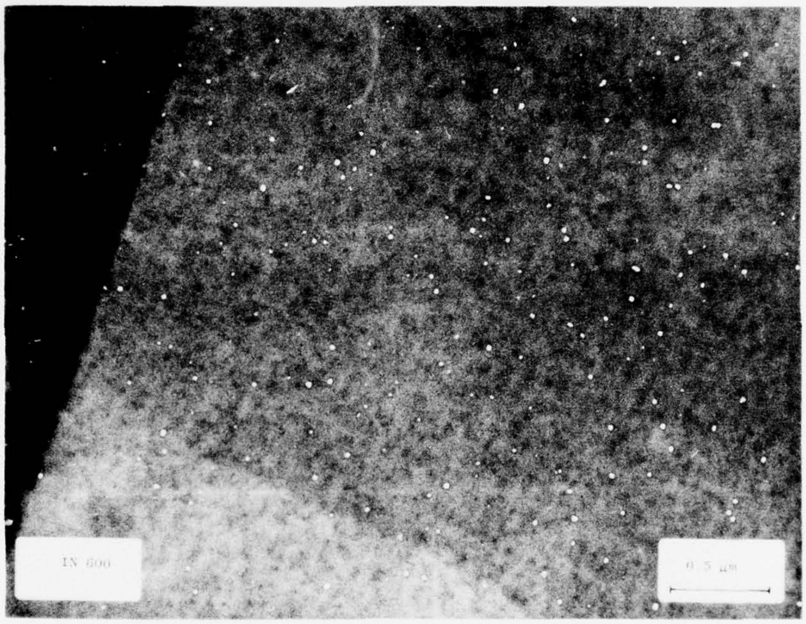

Fig. 5 — Transmission electron micrograph of Inconel 600 irradiated to a fluence of 1.6×10^{22} n/cm^{22}, E > 0.1 MeV, at 455°C (850°F) showing homogeneous distribution of voids and small precipitates which appear as black spots

Inconel 625

Inconel 625 is a nickel-base alloy with small additions of aluminum and titanium which provide some strengthening by γ'. The preirradiation heat treatment produced a γ' precipitate structure which was visible under dark-field imaging conditions.

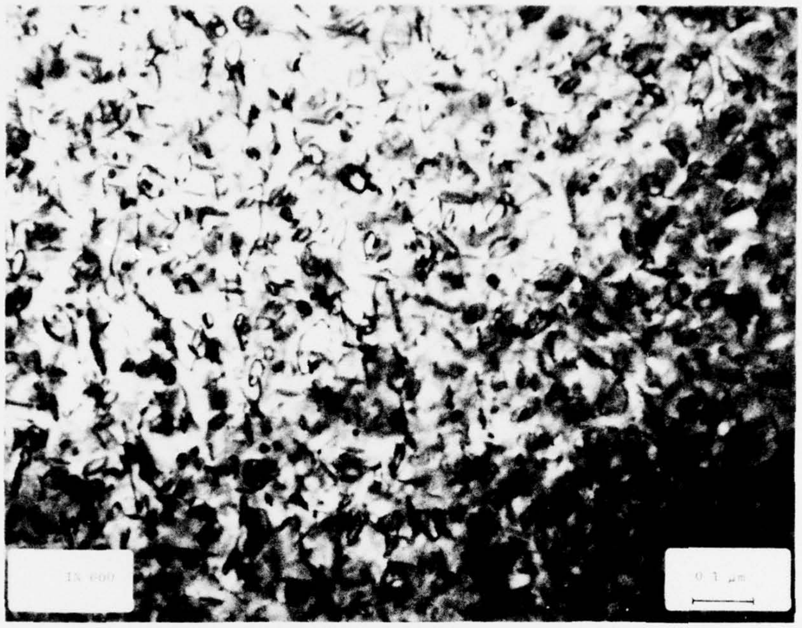

Fig. 6 — Transmission electron micrograph of irradiated Inconel 600 showing radiation-induced dislocation loops having a mean diameter of 37.5 nm.

Irradiation produced a homogeneous distribution of voids, as shown in Fig. 7. The voids had a mean diameter of 11.4 nm and a density of 8.0×10^{14} cm^{-3}, which produced swelling of 0.08%. Small precipitates were observed in the grain interior of the irradiated specimen and appear as black spots in Fig. 7. The γ' precipitate structure after irradiation imaged under dark-field conditions is shown in Fig. 8. Figure 9 shows the dislocation-loop structure produced by these irradiation conditions.

PE-16

PE-16 is a γ'-strengthened nickel-base alloy. Examination of preirradiation heat-treated specimens revealed a uniform distribution of γ' precipitates varying in diameter from 15 nm to 35 nm.

The voids in the irradiated alloy, as seen in Fig. 10, had a mean diameter of 19.4 nm and a density of 6.6×10^{13} cm^{-3}, which yielded 0.03% swelling. No significant increase in the size of the precipitates was noted after irradiation. A high density of dislocation loops with average diameters of approximately 20 nm formed during irradiation, as shown in Fig. 11.

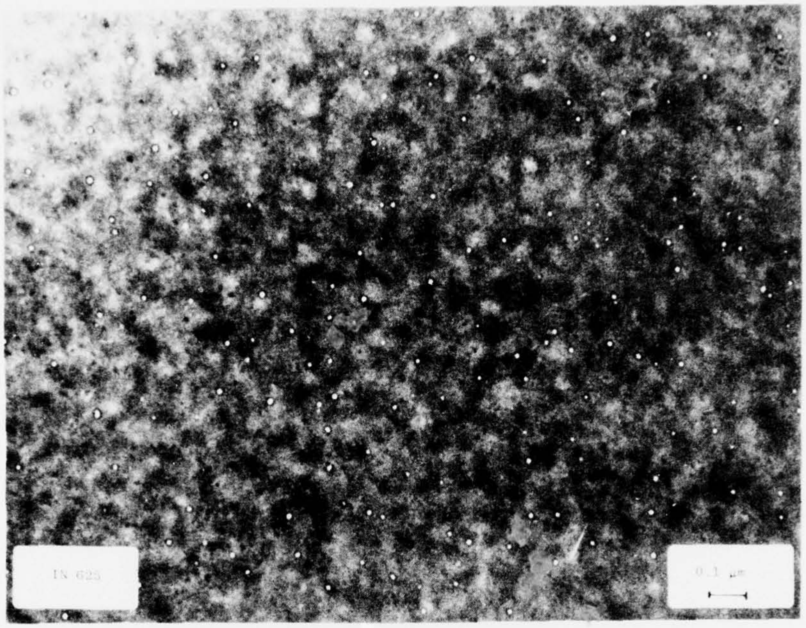

Fig. 7 — Transmission electron micrograph showing voids having a mean diameter of 11.4 nm and small spherical precipitates in Inconel 625 irradiated to a fluence of 1.6×10^{22} n/cm^2, $E > 0.1$ MeV, at 455°C (850°F)

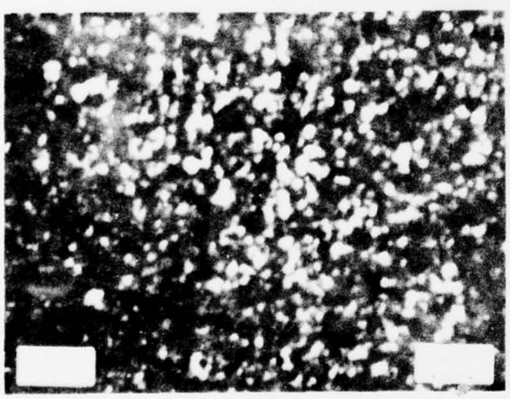

Fig. 8 — Dark-field micrograph of γ' precipitates in irradiated Inconel 625

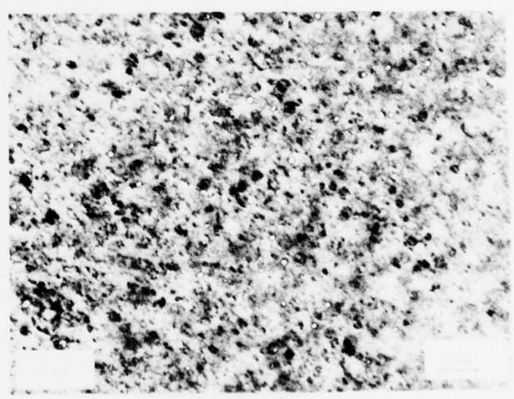

Fig. 9 — Transmission electron micrograph showing radiation-induced dislocation-loop structure in irradiated Inconel 625

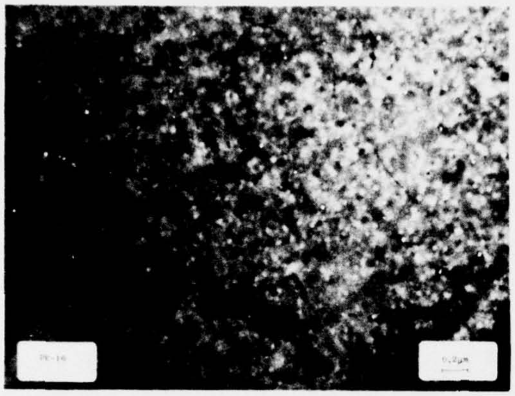

Fig. 10 — Transmission electron micrograph showing void population of PE-16 irradiated to a fluence of 1.6×10^{22} n/cm^2, E > 0.1 MeV, at 455°C (850°F)

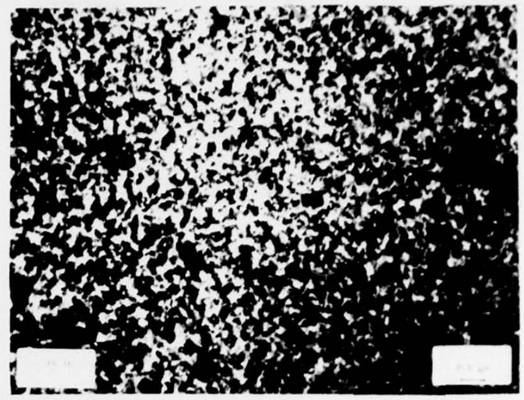

Fig. 11 — Transmission electron micrograph of irradiated PE-16 showing dislocation-loop structure

Sandvik 12R72HV

Sandvik 12R72HV is an austenitic stainless steel with carbide stabilizers added. The prescribed heat treatment was intended to strengthen the material by producing carbide precipitation on dislocations produced by 20% cold work. The precipitation was on a fine scale and difficult to image in the cold-worked material.

The irradiated material had a high density of small voids, many of which appeared to be preferentially aligned in strings, as can be seen in Fig. 12. When the dislocation structure of the same area is imaged (Fig. 13), the alignment of the strings appears to be along the dislocation lines. A number of small precipitates which have formed or coarsened during irradiation can also be seen in Fig. 13. The swelling in this alloy is quite high at 0.3%. The mean diameter of the voids is small, 12.7 nm, but the density is high, 2.6×10^{15} cm^{-3}.

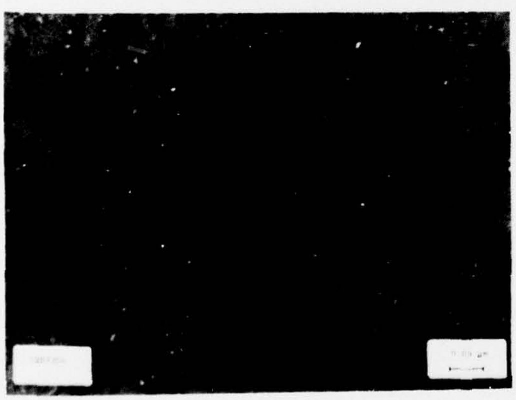

Fig. 12 — Transmission electron micrograph showing a high density of voids aligned in strings in 12R72HV irradiated to a fluence of 1.6×10^{22} n/cm^2, E > 0.1 MeV, at 455°C (850°F)

Fig. 13 — Dislocation structure of the same area as shown in Fig. 12. Comparison of the two micrographs show that the strings appear along the dislocation lines.

Inconel 744

Inconel 744 is a superplastic duplex alloy consisting of small austenite grains in a ferrite matrix. Figure 14 shows the small grain size, 2.28 μm diameter, and unusual banding of the dislocation substructure in this material.

Voids were observed to form in only part of the grains, as shown in Fig. 15. An analysis of a typical area showed that 37% of the area contained voids and 63% did not. This corresponds to the expected ratio of austenite and ferrite, and this identification has been confirmed by electron diffraction patterns from selected grains. The grains which contain voids have a swelling of 2.7%, with a void density of 2.8×10^{15} cm^{-3} and a void mean diameter of 24.6 nm. The remaining grains contained no voids, and when the data are adjusted to reflect the total area, the overall swelling for the Inconel 744 is calculated to be 1%.

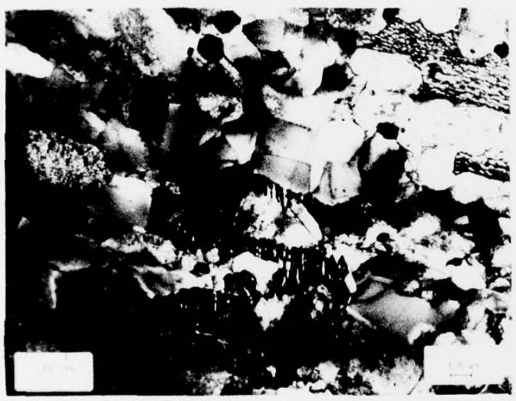

Fig. 14 — Transmission electron micrograph showing small grain size and unusual banding of the dislocation substructure of Inconel 744 irradiated to a fluence of 1.6×10^{22} n/cm^2, $E > 0.1$ MeV, at 455°C (850°F)

Fig. 15 — Transmission electron micrograph of irradiated Inconel 744 showing the high density of voids formed in the austenite grains and the absence of voids in the ferrite grains

The grains which do not contain voids have small clusters of dislocation loops, as shown in Fig. 16. Dark-field TEM techniques were used in searching for precipitates in these grains, but none were found.

DISCUSSION

The alloys surveyed for swelling resistance and alloy stability in this irradiation cover a wide range of commercial alloy compositions across the γ-phase region in a Fe-Cr-Ni ternary diagram as well as one duplex alloy. The alloy compositions are indicated on the Fe-Cr-Ni ternary phase diagram for 650°C (1200°F) shown in Fig. 17.

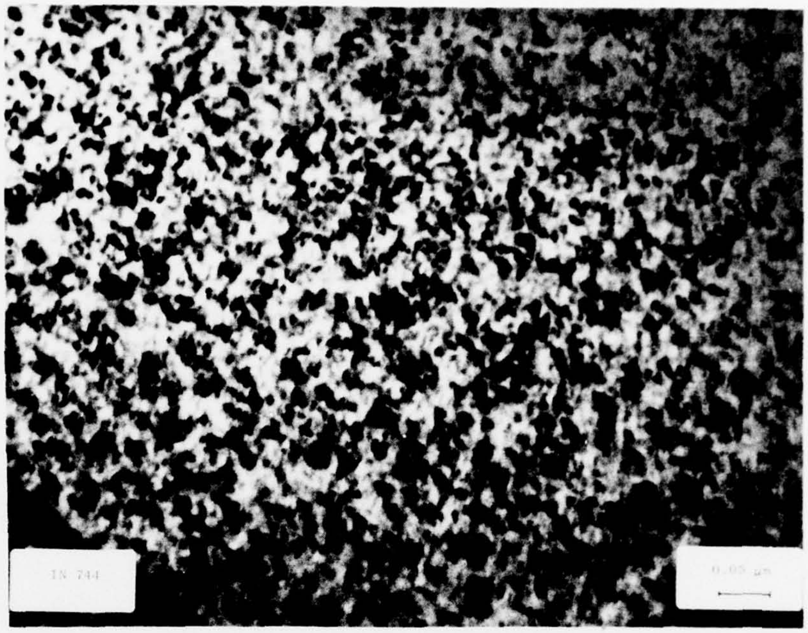

Fig. 16 — Transmission electron micrograph showing small clusters of dislocation loops in the ferritic matrix of irradiated Inconel 744

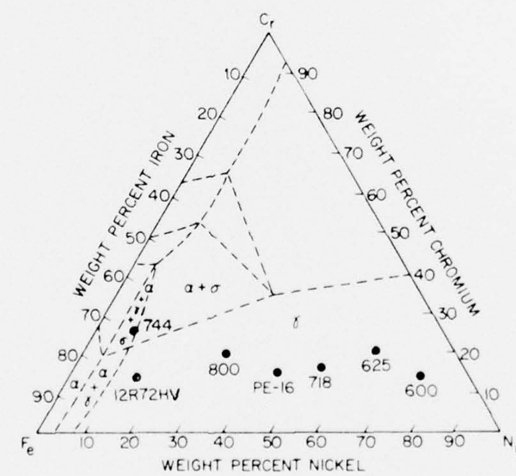

Fig. 17 — Fe-Cr-Ni ternary phase diagram for 650°C (1200°F)

As described in the previous section on results, significant changes were observed in the microstructures of all the alloys examined as a consequence of this irradiation to a fluence of 1.6×10^{22} n/cm^2, E > 0.1 MeV, at 455°C (850°F). These changes in microstructure indicate potential swelling and alloy stability problems. Swelling is of concern, because it can produce dimensional changes in compoent size and shape. None of the swelling values found in this experiment were excessively large, but then the fluence was fairly low for near-core LMFBR applications. Experience with AISI Type 316 stainless steels [1] has shown the swelling rate to increase linearly after an incubation period. Most of the alloys studied here were still in the incubation region. The swelling values observed in Incoloy 800, Sandvik 12R72HV, and Inconel 744 therefore indicate poor swelling resistance can be expected at higher fluences.

Some change in the precipitate phase or distribution was observed in almost all the samples. Changes of this nature can influence the strength and long-term stability of the alloys and hence is also of concern. Since most commerical alloys are metastable, it is not surprising to find precipitate growth under irradiation conditions where the vacancy concentration is increased. Point-defect trapping of solute atoms may also lead to transport and segregation of solutes at interfaces or defect sinks. Some changes are less expected, such as the appearance of "new" phases under irradiation [2]. Damage to the γ' strengthening phase in nickel-base alloys, which results in changes in precipitate size and distribution, has also been observed [3].

The present discussion will summarize the physical metallurgy for these alloys to put the changes observed into perspective and to note potential problems. In many cases the heat treatments used in this experiment differed somewhat from that of commerical practice. These differences will be noted.

Inconel 718

Inconel 718 is a nickel-base alloy strengthened by γ'' and γ'. The literature on the physical metallurgy of the alloy has recently been reviewed and summarized [4], so no effort to provide a complete bibliography will be made. The presence of 0.4A1 and 0.9Ti produce some γ', Ni_3 (A1,Ti), but the primary strengthening phase is γ'' body-centered-tetragonal Ni_3Nb. Commercial heat treatments normally consist of either (1) a solution anneal at 980°C (1800°F) for 1 hr followed by air cooling to 720°C (1325°F), aging at 720°C for 8 hr, furnace cooling for 20 hr to 620°C (1150°F), aging at 620°C for 8 hr, and air cooling or (2) a solution anneal at 1050°C (1925°F) for 1 hr followed by air cooling to 760°C (1400°F), aging at 760°C for 10 hr, furnace cooling at 55°C/hr to 650°C (1200°F), aging at 650°C for 8 hr, and air cooling. Heat treatment 1 may produce a Laves phase (Ni, Fe, Cr)$_2$(Nb, Mo, Ti), which is harmful to ductility and ties up strengthening elements. Heat treatment 2 avoids the Laves phase but probably forms NbC along the grain boundaries. Both heat treatments produce γ' and γ'', and long aging converts some of the γ'' to a more stable orthorhombic form of Ni_3Nb.

The heat treatment received by the material in the H-5 experiment consisted of a solution anneal at 1200°C (2190°F) for 1 hr, air cooling to 790°C (1455°F), aging at 790°C

for 10 hr, air cooling to 650°C (1200°F), aging at 650°C for 10 hr, and air cooling. This heat treatment probably produced some NbC at grain boundaries and somewhat greater conversion of γ'' to Ni_3Nb than the commercial heat treatments. None of these differences are significant.

No attempt has been made to identify and analyze all the phases present in the Inconel 718 used in this experiment. A qualitative comparison of the microstructural features with those described in the literature [5] confirms the presence of γ' and γ''. In the irradiated specimen the presence of γ'' was confirmed by imaging the precipitates in dark field with a superlattice reflection. There were no immediately obvious changes in the precipitate morphology in this Inconel 718 as a consequence of irradiation. Another irradiation of the same material at 748°C to a fluence of 9.0×10^{21} n/cm^2, E > 0.1 MeV, showed a more obvious coarsening of the γ'' precipitates.

Inconel 718 is one of the alloys being extensively studied in the National Alloy Development Program for cladding and duct applications, and extensive high-fluence data should soon be available from that program. Ion bombardment of Inconel 718 has been found to accelerate the aging of the metastable γ' and γ'' phases to form the η phase, Ni_3Ti [6].

The swelling resistance of Inconel 718 appears to be quite good, with only a few scattered voids found in these specimens, for a swelling of 0.02%. A previous irradiation experiment on Inconel 718 at 540°C and a fluence of 7.4×10^{21} n/cm^2, E > 0.1 MeV, showed 0.1% swelling [7]. Another specimen irradiated at 748°C to a fluence of 9.0×10^{22} n/cm^2, E > 0.1 MeV, had no voids.

Incoloy 800

Incoloy 800 is a solid-solution-strengthened 30Ni-20Cr austenitic alloy. Recently produced heats of Incoloy 800 have small additions of aluminum and titanium to provide some γ' strengthening. The heat used in the H-5 experiment however was an older version of Incoloy 800 which did not have the titanium and aluminum additions.

The usual commercial heat treatment is a solution anneal at 1120 to 1150°C (2050 to 2100°F) for 1 to 2 hr. This produces a few titanium cyanonitride precipitates that are stable up to the melting point. Chromium carbides form in the 540 to 1090°C (1000 to 2000°F) range and can lead to sensitization to corrosion in the 540 to 760°C (1000 to 1400°F) range. In the heats containing aluminum and titanium, γ' will form during long-term aging in the 565 to 595°C (1050 to 1100°F) range [8]. Titanium cyanonitride, $M_{23}C_6$, and γ' have been reported in a fatigued specimen of Incoly 800 [9].

Heat treatment given to the material in the H-5 experiment consisted of a solution anneal at 1200°C (2100°F) for 1 hr, air cooling to 790°C (1455°F) aging at 790°C for 14 hr and then air cooling. This heat treatment was intended to precipitate the γ' to minimize changes in properties during irradiation and was selected before it was realized that the material used in this experiment was an older heat not containing aluminum and titanium. A few large globular precipitates were present in the specimen after this annealing treatment, but no other precipitates were visible.

Following irradiation a high density of rod-shaped precipitates was uniformly dispersed throughout the matrix. A similar precipitate was also observed in Incoloy 800 irradiated at 540°C to a fluence of 7.4×10^{21} n/cm^2, E > 0.1 MeV, in a previous experiment [7]. The precipitate has not been identified at this time.

Swelling in Incoloy 800 in this experiment was 0.26%, one of the higher values observed among these alloys. The previous irradiation experiment at 540°C and 7.4×10^{21} n/cm^2, E > 0.1 MeV, produced 0.064% swelling. This is appreciable swelling for such a low fluence, thereby indicating Incoloy 800 has poor swelling resistance. The peak swelling temperature for Incoloy 800 is estimated to be between 450 and 550°C.

Inconel 600

Inconel 600 is a solid-solution-strengthened 15Cr-8Fe nickel-base alloy [10]. The commercial heat treatment consists of a solution treatment at 1120°C (2050°F) for 12 hr followed by air cooling. This produces a few large titanium carbonitride precipitates randomly dispersed in the material. Chromium carbides precipitate in the range 540-to 980°C (1000 to 1800°F) both at grain boundaries and in the matrix. This may leave the material sensitized to corrosive attack if aged in the range 540 to 760°C (1000 to 1400°F). Predominant carbides are Cr_7C_3 and $M_{23}C_6$.

The heat treatment given the Inconel 600 used in the H-5 experiment consisted of a solution anneal at 1200°C (2190°F) for 1 hr, air cooling, and an aging treatment at 790°C (1455°F) for 4 hr and air cooling to precipitate the carbides. This produced carbide precipitation along the grain boundaries, but no precipitates were observed in the matrix. Irradiation produced additional precipitation in the form of a fine dispersion of platelets. The dislocation structure produced by this irradiation is in the form of loops. Small spherical precipitates which have not been identified are also observed on these loops.

Swelling in Inconel 600 under these conditions is 0.04%. The voids are homogeneously distributed and do not appear to be associated with the precipitates. The aging treatment may have removed some elements from solid solution that would contribute to swelling resistance. Another experiment without the second aging treatment would be of interest to compare the effect of heat treatment on both swelling resistance and alloy stability.

Inconel 625

Inconel 625 is a 20Cr-3Fe nickel-base alloy with additions of 9Mo and 3.5Nb to provide solid-solution strengthening [11]. Additions of aluminum and titanium also lead to some formation of γ' but not in large enough quantities to influence the hardening. The most commonly used heat treatment is a solution anneal at 1040 to 1200°C (1900 to 2200°F) for 1 to 2 hr [12]. A modified heat treatment that provides higher strength includes an intermediate anneal at 1040°C (1900°F) for 1 hr air cooling, aging at 540 to 700°C (1000-1300°F) for 16 hr and air cooling [12]. The complex composition of this alloy leads to a variety of phases, many of which depend on the heat treatment employed. A comprehensive study of precipitation behavior in this system has been reported by Schnabel et al. [13].

The treatment given the material in the H-5 experiment consisted of (Table 2) solution treatment at 1200°C (2190°F) for 1 hr, air cooling, and double aging at 790°C (1450°F) for 10 hr and at 650°C (1200°F) for 10 hr. An analysis of the TTT diagrams for Inconel 625 [18] suggests this heat treatment would produce a mixture of $M_{23}C_6$ with lesser amounts of M_6C, of MX, which is mostly Mn(CN), and of γ'', which is body-centered-tetragonal Ni_3Nb. A small amount of the stable orthorhombic Ni_3Nb phase or η Ni_3Ti might possibly form at longer aging times and somewhat higher temperatures. The microstructure of the unirradiated Inconel 625 showed extensive precipitation along the grain boundaries, probably $M_{23}C_6$, but no pr[illegible]itates were readily visible in the matrix. High-magnification photomicrographs gave some h[illegible] contrast around a coherent phase, most likely γ''.

[illegible] of the specimen following irradiation showed extensive precipitation in the for[illegible]elets or rods and spherical precipitates on irradiation-produced dislocation loops. A[illegible]d structure was observed and readily imaged in a dark field with the superlattice reflection. It occupies a fairly large volume fraction in the structure and is probably γ'', although not positively identified yet.

Swelling in Inconel 625 was 0.08%, nearly the same as swelling in Inconel 600, which has approximately the same major-component composition. A comprehensive effort to identify the precipitated phases and kinetics under irradiation should be mounted it Inconel 625 is to be used in FBR service.

PE-16

PE-16 is a 16Cr-43Ni-35Fe alloy strengthened by γ', which is $Ni_3(Al,Ti)$. The alloy is included in the National Alloy Development Program for LMFBR cladding and duct applications. More extensive information on stability under irradiation and swelling at high fluences will soon be available. The recommended heat treatment for PE-16 includes a solution treatment at 1040°C (1900°F) for 2 to 4 hr followed by double aging between 700 and 900°C (1290 and 1650°F) to precipitate γ'. The vendor recommended a treatment of 800°C (1475°F) for 2 hr and 700°C (1290°F) for 16 hr with air cooling. Sklad et al. [14] have recently reported a study on PE-16 which employed two heat treatments: (a) 1040°C for 2 hr, 800°C for 2 hr, and 700°C for 16 hr with air cooling and (b) 1040°C for 4 hr, 900°C for 1 hr and 750°C for 8 hr with air cooling. Treatment (a) produced γ' with a mean diameter of 22 nm, and treatment (b) produced precipitates with a mean diameter of 10 nm.

The heat treatment employed for the material in the H-5 experiment was devised without benefit of data sheets on optimum strengths and was intended simply to produce the approximate phases. A typical superalloy heat treatment was used consisting of a solution anneal of 1200°C (2190°F) for 1 hr followed by aging at 790°C (1455°F) for 4 hr and air cooling. This heat treatment produced γ' with a somewhat larger diameter of 25 nm.

Irradiation produced no notable change in the γ' precipitate structure. A fine dispersion of precipitates did form during irradiation however, with some in the matrix, some attached to the irradiation-produced dislocation loops, and some associated with voids. In the work reported by Sklad et al. [14] PE-16 was irradiated at several temperatures to fluences up

to 4.0×10^{22} n/cm^2, E > 0.1 MeV. Specimens irradiated at 450°C, as in the present experiment, were reported to show some redistribution of γ' onto dislocation loops produced during the irradiation and some coating of voids.

Swelling in the PE-16 was measured at 0.03%. This value agrees well with that found in the Sklad study [14] for comparable temperatures and fluences: 0.02% for heat treatment (a) and 0.03% for heat treatment (b). The heat treatment seems to influence the temperature dependence of swelling at higher fluences. Heat treatment (a) gave maximum swelling at 500°C, and heat treatment (b) seemed to produce a plateau from 425°C to 500°C and only half the swelling at 500°C as heat treatment (a). Additional studies are required to map the alloy stability and swelling response of PE-16 at high fluences if the material is to be used in FBR structural applications.

Sandvik 12R72HV

Sandvik 12R72HV is a 15Cr-15Ni austenitic stainless steel with 1.2Mo for solid-solution hardening and 0.5Ti for precipitation hardening [15]. The steel is made by vacuum melting by the consumable-arc process. The heat treatment recommended consists of a solution anneal at 1150°C (2100°F) for 1 hr, air cooling, followed by light cold working of 10-20%, and an age at 700 to 900°C (1300 to 1650°F) to produce precipitation of titanium carbonitrides on the dislocation network. $M_{23}C_6$ also forms at the grain boundaries during this heat treatment.

This practice was followed for the heat treatment of the Sandvik 12R72HV used in the H-5 experiment. A solution anneal at 1200°C (2190°F) for 1 hr with air cooling, was followed by 20% reduction in thickness by cold rolling, aging at 850°C (1560°F) for 4 hr, and air cooling. This heat treatment led to precipitation of $M_{23}C_6$ along the grain boundaries and a fine dispersion of precipitates on the dislocations produced by the cold-worked structure.

Irradiation coarsened the precipitates on the dislocation structure but caused no other major changes. A high density of voids with an unusual elongated morphology was produced by this irradiation. Many of the voids appear in strings along dislocation lines. It appears that either certain elements in atmospheres near dislocations favor nucleation of voids or these elements redistribute onto the voids and reduce the surface energy so that the voids grow preferentially along the dislocations. Swelling of 0.3% in this alloy was quite high for the fluence. Other irradiation data on Sandvik 12R72HV have been reported by Atomics International [16] for 500°C and a fluence of 4×10^{22} n/cm^2, E > 0.1 MeV. A foil specimen given only a solution anneal showed 1.8% swelling from TEM measurements, and a tensile specimen given an aging treatment showed a change in immersion density of 1.1%. These experiments indicate Sandvik 12R72HV has poor swelling resistance and is not likely to be suitable for extended LMFBR service.

Inconel 744

Inconel 744 is an 8Ni-27Cr-Fe base alloy with a duplex structure consisting of a fine dispersion of austenite grains in a ferrite matrix [17]. With proper heat treatment the

material is superplastic. It was included in this experiment to survey the response of such a structure to irradiation and not because of any potential FBR applications. Heat treatment suggested by the vendor included a solution anneal at 1050°C for 15 min, cold work, an anneal at 815°C (1500°F) for 1 hr, and a water quench followed by another anneal between 425 and 540°C (800 and 1000°F) [17].

The thermomechanical treatment employed in this experiment consisted of a solution anneal at 1050°C (1925°F) for 15 min, a 50% reduction in thickness by cold rolling, and anneals at 870°C (1600°F) for 1 hr and at 455°C (850°F) for 3 min and air cooling. This treatment produced a fine-grained structure with a few globular precipitated randomly scattered through the structure. The σ phase is also expected to form in this alloy upon aging at temperatures below 700°C (1290°F) for longer than 1 hr. Aging for 100 hr will produce up to 50% σ phase [17].

Irradiation produced a most unusual response in this material: high swelling in the austenite phase and no swelling in the ferrite phase. The swelling of 2.8% observed in the austenite phase is extremely high for a commerical-purity Fe-Cr-Ni alloy. The ferrite grains on the other hand had no voids at all and a dislocation structure consisting of small loops just barely resolvable and a high density of unresolvable loops appearing as black spots. An average of swelling over th entire specimen gave about 1%, a value which is still extremely high for this fluence for a commercial alloy.

GENERAL COMMENTS ON SWELLING TRENDS

The alloys surveyed in this study cover a wide range of nickel compositions in the γ field of the Fe-Cr-Ni phase diagram. Johnston et al. [18] surveyed this same region for swelling resistance using ion bombardment to produce the damage. They found a strong correlation between swelling resistance and nickel content, with the lowest swelling at nickel contents above 35%. Many of the alloys used in their survey were also used in the present study, and in general there is close agreement with the trends observed. Watkin [19] reported a similar survey of swelling resistance in commercial alloys after irradiation in the Dounreay fast reactor (DFR) to fluences of 2.26 and 5.02×10^{22} n/cm^2, $E > 0.1$ MeV. Swelling data were reported for an irradiation temperature of 600°C and compared with several correlation parameters such as equivalent nickel content, percent σ phase, and electron vacancy concentration. A low-swelling region was observed after neutron irradiation in the same composition range as observed by Johnston et al. by ion irradiation [18].

The results from the present experiment show good agreement with these surveys. The swelling resistance of Inconel 600, Inconel 625, PE-16, and Sandvik 12R72HV, which were examined in the ion-bombardment experiment [18], were found to closely parallel those trends in the current neutron irradiation. The general compositional trends observed after 600°C irradiation in the DFR [19] were also apparent in the current experiment. PE-16 was the only alloy common to all three studies and was found to be at or near the lowest swelling in all three studies. Inconel 718, which was not included in either of the other studies, exhibited slightly lower swelling than PE-16 and is in the same composition range. The Incoloy 800 appeared to have less swelling resistance than Johnston et al. [18] found, and this may be a

reflection of the absence of titanium and aluminum additions in the heat used in this study. Finally, the austenite phase in the Inconel 744 exhibited the highest swelling rate that has been reported for any Fe-Cr-Ni alloy. A linear extrapolation to the fluences in the DFR experiment [19] would give a swelling value of 9.3% as compared with 4.5% for AISI type 316 stainless steel and 6% for FV 548.

CONCLUSIONS

Swelling resistance and alloy stability under neutron irradiation have been surveyed in seven commercial alloys of potential interest for FBR structural materials. Inconel 718, Incoloy 800, Inconel 600, Inconel 625, PE-16, Sandvik 12R72HV, and Inconel 744 were irradiated in EBR-II to a fluence of 1.6×10^{22} n/cm^2, E > 0.1 MeV, at 455°C. The following observations and conclusions were reached:

- Inconel 718 and PE-16 exhibited the best swelling resistance.
- Sandvik 12R72HV and Incoloy 800 exhibited the poorest swelling resistance of the common commerical alloys.
- Inconel 744, and experimental superplastic alloy with a duplex microstructure, exhibited one of the highest swelling rates known for the austenite phase but showed no swelling in the ferrite phase.
- Inconel 625, Inconel 600, and Incoloy 800 showed extensive precipitation of phases not present prior to irradiation. These reactions should be studied in greater detail to ascertain the kinetics of the processes and to determine the effects of this precipitation on mechanical properties, if these alloys become leading candidates for FBR structural materials.

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REFERENCES

1. F.A. Garner et al., "Development and Evaluation of a Stress-Free Swelling Correlation for 20 Percent Cold-worked Type 316 Stainless Steel," ASTM STP 611, pp. 208-226.

2. J.A. Hudson, "Structural Stability During Irradiation," J. Br. Nuclear Energy Society 14, 127-136 (1975).

3. L.G. Kirchner et al., "Nickel Ion Damage in a Precipitation Hardened Nickel-Aluminum," ASTM STP 611, pp. 370-384.

4. M.L. Bleiberg, R. Bajaj, I.M. Baron, A. Chang, R.W. Chickering, and S. Diamond "Swelling and Gamma-Prime Particle Stability of Ion-Bombarded Iron-Chromium-Nickel Alloys," ASTM STP 611, pp. 315-336.

5. D.D. Keiser, and H.L. Brown, "A Review of the Physical Metallurgy of Alloy 718," ANCP-1292.

6. W.L. Bell et al., "Microstructural Changes in Ion-Irradiated Commerical Alloys," ASTM STP 611, pp. 353-369.

7. F.A. Smidt, Jr., and J.R. Reed, "Examination of Microstructure of Inconel 718, Incoloy 800, PH 13-8 Mo, Molybdenum and Niobium After Irradiation," pp. 11 through 21 in NRL Memorandum Report 3010, Feb 1975.

8. Inconel Alloy 800 Bulletin, Huntington Alloy Products Division, The International Nickel Company, Inc., 3rd edition, 1969.

9. Hong Nahm and J. Moteff, "Second Phase Formation and its Influence on the Fatigue Properties of Incoloy 800 at Elevated Temperatures," Metallurgical Transactions 7A, 1473 (Sept. 1976).

10. Inconel Alloy 600 Bulletin, Huntington Alloy Products Division, The International Nickel Company, Inc., 3rd edition, 1969.

11. Inconel Alloy 625 Bulletin, Huntington Alloy Products Division, The International Nickel Company, Inc., 3rd edition, 1976.

12. Aerospace Structural Metals Handbook, Mechanical Properties Data Center, Belfour Stulen, Inc., Traverse City, Mich., Vol. 4, 1975.

13. E. Schnabel, H.J. Schüller, and P. Schwaab, "The Precipitation and Recrystallization Behaviour of the Nickel-Base Alloy Inconel 625," Praktische Metallographie 8 (No. 9), 521-527 (Sept. 1971), in German and English.

14. P.S. Sklad et al., "Effects of Neutron Irradiation on Microstructure and Mechanical Properties of Nimonic PE-16," ASTM STP 611, pp. 139-155.

15. L. Egnell, "An Austenitic Steel for High-Temperature Service Applications," Metal Progress 98 (No. 3), 102-107 (Sept. 1970).

16. K.R. Garr, A.G. Pard, and D. Kramer, "The Effect of Neutron Irradiation on Types 316, 321, and Sandvik 12R72 Stainless Steels," AI-AEC-13130 Report, Atomics International Division, Rockwell International, June 1974.

17. Inconel Alloy 744 Bulletin, Huntington Alloy Products Division, The International Nickel Company, Inc.

18. W.G. Johnston, J.H, Rosolowski, A.M. Turakalo, and T. Lauritzen, "An Experimental Survey of Swelling in Commercial Fe-Cr-Ni Alloys Bombarded with 5-MeV Ni Ions," J. of Nuclear Materials 54, 24-40 (1974).

19. J.S. Watkin, "Dependence of Void Swelling on the Electron Vacancy Concentration," ASTM STP 611, pp. 270-283.